RESEARCH

USE OF BIOMASS CONVERSION PRODUCTS AS BINDERS FOR FINE COAL AGGLOMERATION

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1.0 SUMMARY

This report presents results of a program to investigate use of unseparated biomass conversion products as binders for fine coal agglomerates.

Raw products of biomass conversion processes are generally three phase (oil - aqueous - insoluble) mixtures with varying degrees of emulsion stability. In the raw state, fuel use of these products is limited to open combustion in large burners, and - except for pumpability - the products are only marginally superior to biomass feedstock. Refining of raw conversion products is difficult and expensive, so a market for the raw product is desireable.

Agglomeration of fine coal is a major problem, particularly for Rocky Mountain coals. Petroleum oils can be used for this purpose, and - to the extent that unseparated biomass conversion products can substituted for oil - this bulk use could provide a market for biomass derived "liquid" products without need for costly separation/refining.

The test program included investigation of three agglomeration technologies; NRC oil agglomeration, Dravo pressure filtration/forming, and pin agglomeration ("turbulation"). The former two are methods for both dewatering and agglomeration; turbulation is a method for micro-agglomeration/bonding of dry fines such as are produced from fluid bed thermal drying of coal.

Three types of biomass derived oils were used in testwork: vacuum pyrolysis oil (Laval University), a slurry hydrogenation product (Albany TR-12 USDOE), and steam pyrolysis products produced in-house.

Only the Albany oil was available in bulk, so oil - biomass oil comparison work was concentrated on this product. Product from the Waterloo flash pyrolysis program was not available for the study. Cold Lake pitch, No. 6 fuel oil and diesel (No. 2 oil) were used for comparison studies.

Coals used were from Bryon Creek Collieries Limited (thermal coal flotation concentrate), Fording Coal Limited (fines from blended product) and Westar Coal Limited (metallurgical coal fines; dryer dust from the Greenhills Mine). Sulphite lignin was also used in tests on dry coal fines.

In oil agglomeration tests, all biomass oils performed poorly. Selectivity of coal - ash separation was negligible, presumably due to the hydrophilic nature of biomass oils. Supplemental tests using the Laval oil as a froth flotation reagent also showed poor selectivity.

In Dravo pressure filtration/forming tests, the Laval and Albany oils performed reasonably well compared to petroleum oils but showed no advantages in dose requirement or properties.

Pin agglomeration tests with emulsified Laval oil materials produced results which were qualitatively comparable to those obtained with No. 6 fuel oil at a higher addition level. For the Laval product, good results were obtained by using a 30% oil-water emulsion at 3% (biomass) oil addition, compared to 6% for No. 6 fuel oil. This is potentially significant, since fuel oil is not cost effective at the required dosage compared to sulphite lignin. Lignin products are effective at ~1% addition level but have a negative effect on coking properties (free swelling index, coke strength). Coking properties were either unaffected or marginally improved with the biomass oil binder. Pitch could not be effectively dispersed in the pin agglomerator.

In summary, the only application in which a biomass product provided equivalent or better performance compared to petroleum oils was in pin agglomeration of dry fines. In this application, the Laval vacuum pyrolysis oil provides satisfactory bonding with no reduction in coking properties.

This potential use, while somewhat specialized, is potentially significant to Western Canada producers of bituminous coals. All of the metallurgical coal producers have thermal dryers, and - to varying degrees - all have experienced problems relating to relatively high fines contents. If biomass derived oil with the performance characteristics of the Laval vacuum pyrolysis product were available, it would be preferred to lignin binder. Cost of bulk lignin delivered to southeast B.C. sites is in the range of $(Can) 0.50-0.60/Kg, or $5-6/tonne of coal treated at 1% addition level; at the 3% level required for the Laval product, a price of $0.20/Kg would make the material cost competitive with lignin, and its compatibility with coking would make it the preferred material, possibly even at a higher price. Since the $0.20/Kg inferred price for the product is roughly equivalent to the current cost of fuel oil (on a weight basis), it would appear that there is at least one potential coal agglomeration technology for which raw biomass derived oil could be marketed at parity with current oil prices. The size of the potential market for fine coal binder in Southeast B.C. would allow production of biomass liquids at a level of 30-50 tonnes/day.

It is recommended that staff of the Bioenergy Development Program review information on the Laval and Waterloo pyrolysis processes to determine whether production would be feasible at the indicated scale of operation and product price. If feasibility is indicated further necessary technical work would be justified.

2.0 INTRODUCTION

2.1 BACKGROUND

2.1.1 Biomass Derived Liquids

In response to abrupt oil price increases in the 1970's, and perceived shortfalls in oil supplies, there was a surge of effort to develop alternative sources of liquid fuels. Biomass - particularly where it is available as "waste" due to lack of markets for low grade solid fuel - is a potentially attractive feedstock for liquid fuel production.

In Canada and the USA, where forestry and agricultural industries generate suitable quantities of such waste, research programs were undertaken to develop technology for conversion of biomass to liquid fuel products which could be economically transported and utilized.

In Canada, these efforts - largely supported by the ENFOR Biomass Conversion Program - resulted in development of several "processes". One, the Waterloo flash pyrolysis program was identified by an International Energy Agency study [1] as having low production costs, at least relative to other "processes" at similar stages of development. A vacuum pyrolysis process is at a similar stage of development at Laval University. In the USA, larger scale pilot plant tests were done on slurry hydrogenation. This method produces a different material from pyrolysis and - partly because it is available in bulk - it has been used in a variety of test programs.

B.C. Research has also investigated biomass conversion [2] using supercritical extraction with organic solvents; consideration of the requirements for a product to be used in coal binding (see below) suggested that this approach might be simplified using water as the fluid medium, since it is not essential to separate the coproduct char from water insoluble oil.

All biomass liquefaction programs have suffered funding difficulties in the current period of low petroleum prices and perceived high availability.

This study on use of biomass conversion products as coal binders was undertaken to investigate whether these products could - if commercially available - find a market niche, preferably at oil equivalent or higher prices, which would be required to justify investment in demonstration and commercial scale plants.

2.1.2 Coal Agglomeration

In Western Canada, bituminous coals from the Rocky Mountain area are mined primarily for export as metallurgical (coking) coal. Due to tectonic disturbance, virtually all of these coals generate large quantities of fines during handling and processing. These fines (generally defined as 0.5mm x 0) and especially the 0.15mm x 0 "dust" (although it is often referred to in less salubrious terms), cause significant problems in handling, bin flow and airborne dust loss through the complex transport system required to deliver them to worldwide markets. Fines also cause processing problems in dewatering/drying, although these have been largely solved - at a cost - by adaptation of commercial technologies.

In this study, three agglomeration technologies have been investigated: NRC oil agglomeration, Dravo filtration/forming and pin agglomeration (turbulation). The first two, as noted in the project proposal [3] offer potentially attractive prospects for dewatering of coal fines and - for oil agglomeration - rejection of ash, as well as producing handleable, dust free materials. The pin agglomeration work was only undertaken when test results indicated poor performance of biomass derived materials relative to petroleum products in the two processes, initially investigated.

2.1.2.1 Binders for Coal Agglomeration

The key property of coal binder is ability to hold a multiplicity of fine particles together in larger groups (agglomerates) which are sufficiently stable to survive mechanical handling, rewetting and air drying during loading, unloading and open stockpiling. Other important attributes of coal binders include low cost, compatibility with end use and safety (including environmental safety).

For dewatering (Dravo process) and beneficiation/dewatering (oil agglomeration) the binder must also - at least largely - be water insoluble and hydrophobic to avoid loss in aqueous effluent from dewatering.

2.2 OBJECTIVES

Objectives of the study as outlined in the project proposal included:

- preparation of aqueous thermolysis products from biomass.
- evaluation of unseparated biomass conversion products for coal evaluation using spherical agglomeration and Dravo filtration/forming.

When it became apparent that available biomass products were not attractive relative to petroleum oils for these agglomeration technologies, pin agglomeration of dry coal fines was also investigated.

3.0 MATERIALS AND METHODS

3.1 COAL SAMPLES

Fine coal samples for agglomeration tests were from Byron Creek Collieries Limited, Fording Coal Limited and Westar Mining Limited. The Byron Creek material was froth flotation concentrate from reground gravity rejects [4]; this material is a low volatile thermal coal.

The Fording Coal fines were prepared by wet separation of -100 mesh fines from a bulk sample of metallurgical coal product.

The Westar sample was cyclone recovered product from thermal drying of metallurgical coal from the Greenhills mine.

3.2 BIOMASS CONVERSION PRODUCTS

Biomass "oils" were obtained from three sources:

- Albany TR-12 "oil" from the USDOE pilot test on hydrogenation of wood [5].
- vacuum pyrolysis product from Laval University.
- atmospheric and high pressure steam pyrolysis products from poplar chips produced in-house by atmospheric pressure pyrolysis in a tube furnace at temperatures up to 350°C and tubing bomb [6] aqueous thermolysis at temperatures in the range of 250-350°C.

Other biomass derived products used in the program included wood pulp fibre (newsprint reduced to fibre in a blender) and ammonia base sulphite lignin ("Orzan A", product of ITT Rayonier Inc., Hoquiem, WA, USA).

3.3 PHYSICAL/CHEMICAL ANALYSES

Coal samples were characterized by:

- proximate analysis using a Dupont thermogravimetric analyzer.
- free swelling index.
- coke strength by tumbler test [7].

Biomass products were characterized by analytical procedures including:

- thermogravimetric analysis (distillables, volatiles, combustibles, ash).
- moisture (ASTM D95-70).
- thermogravimetric simulated distillation.
- elemental analysis (subcontract to Canadian Microanalytical Limited).

3.4 AGGLOMERATION

3.4.1 NRC Oil Agglomeration

Oil agglomeration tests were done with a variable speed turbine mixer, using slurry samples generally heated to 95-98°C (incipient boiling) due to the viscous nature of most biomass and petroleum based binders. In some cases (particularly in tests with the Albany TR-12 oil), extended high speed mixing was used to obtain homogenous dispersion. High and low shear mixing times were determined on an ad hoc basis by pipet withdrawal of small samples for visual examination and sieve separation.

3.4.2 Dravo Pressure Filtration/Forming

Coal-binder slurry samples, generally conditioned at 95-98°C as for spherical agglomeration were pressure filtered in a Dravo designed laboratory test unit comprising an 8cm diameter pressure chamber and a filter platen with 1mm grooves (feed side) tapered to 1.5mm on the filtrate side. Pressure was applied with a Tinius Olsen mechanical tester using manual control of pressure application rate and time at maximum pressure.

3.5 PIN AGGLOMERATION

Pin agglomeration tests were done in a 7.5cm diameter x 30cm tube agglomerator with a shaft holding 36 radially mounted pins and rotating at 1800 rpm.

4.0 RESULTS AND DISCUSSION

4.1 CHARACTERIZATION OF COAL SAMPLES

Proximate analyses and other data on the coal samples used are presented in Tables 1 and 2. The Byron Creek material was ball milled prior to flotation, which accounts for its extreme fineness; the Fording sample is typical of flotation concentrate (~30-40% of net product), and the Greenhills sample is reasonably representative of "dust" from drying operations.

4.2 CHARACTERIZATION OF BIOMASS BINDERS

Results of analyses of biomass conversion product samples are summarized in Table 3.

The Albany TR-12 oil is lowest in oxygen content (14.7%, water free) due to production via slurry hydrogenation. The Laval pyrolysis oil at 25.9% oxygen (water free) is also substantially lower in oxygen than other materials tested and is the only sample with a pour point near ambient temperature. Solvent extracts (normally THF) from the Laval oil and steam pyrolysis products are fluid at ambient temperature but char contents of unseparated products are high enough to render them essentially solid at ambient temperature.

Of the aqueous thermolysis products, only the material produced at 300°C was considered sufficiently converted for use in agglomeration testing; lower temperature resulted in very low extractable liquid yields. The char content of atmospheric steam pyrolysis was disappointingly high; results of other studies [8] suggest that a high rate of heating to the finish temperature (350°C) would improve tar yield and correspondingly reduce char formation.

4.3 PETROLEUM BINDERS

Properties of petroleum base binders are presented in Table 4. The most dramatic difference is in pour point, with the Cold Lake pitch being semi-solid at ambient temperature. However, all have adequate fluidity in the 95-100°C ranged used for conditioning of coal-binder slurries.

4.4 DRAVO PRESSURE FILTRATION/FORMING

4.4.1 Devatering

The potential attraction of the Dravo process arises from combining of fine coal devatering and agglomeration in a single operation. Results presented in Table 5 indicate that devatering of fine coal is less effective with Laval and Albany biomass conversion products than with petroleum oils and marginally less effective than with fibre additive only.

In the 3% additive range (probably an economic upper limit for oil), as formed moisture contents were best for pitch and No. 6 oil (19% and 16.5% respectively), marginal for the Laval and Albany oils (22.1%, and 23%) and poor for the steam pyrolysis products (28-32%). The latter result is presumably due to the high char/low oil content of these products.

These data indicate that acceptable moisture levels can be obtained with biomass oils, but there is no significant moisture reduction compared to use of fibre alone as a filter aid.

It should be noted that overall product moisture contents of clean coals are limited to 8%, but the -150μ fines (normally 15-24% of total product) are the wettest component of feed to thermal drying. If they were processed separately, even to 20-22% moisture. mechanical/thermal devatering of the +150μ fraction to moisture contents in the 2-3% range

would result in an acceptable overall moisture in final product and avoid overdrying of fines, thus minimizing dust generation.

4.4.2 Mechanical Stability

Due to the multiple rehandling in coal transport, formed agglomerates must be mechanically strong enough to prevent breakdown to fines.

Several test procedures for mechanical stability were evaluated, including crushing strength and tumbling tests, before settling on the following procedures which is considered to be a reasonable laboratory approximation of actual handling conditions:

- drop a single 400-500g agglomerate from a 1m height into a steel tray.
- sieve at 6mm, retain undersized and drop oversize again, repeating to a total of five drops.
- weigh undersize, sieve 100g for 2 minutes (rotap), record overall size distribution (+6mm, 6x2mm, 2x0.5mm, 0.5x0.15mm, -0.15mm).

This procedure shatters all pressure filtered agglomerate products.

Results presented in Table 6 indicate that fines generation from agglomerates is satisfactory when tested at the as formed moisture content. In the dry condition, however, substantial differences in stability are observed.

For pitch, No. 6 oil and Albany oil, fines (<0.15mm) were all $\leq$10% whereas the Laval oil generated 26% fines and steam pyrolysis product 44% fines.

Since air exposed surfaces of stockpile and rail can approach dryness, the data indicate that - of the biomass products - only the Albany oil shows performance comparable to petroleum products.

4.4.3 Moisture Resistance

Oven dry agglomerates were tested for moisture resistance by 24h water immersion; the sample was then reweighed to determine moisture pickup. Somewhat surprisingly, this procedure indicated very little difference between petroleum and biomass based binders at the 3% addition level. It would appear that - although the biomass materials are less oleophilic - they are effective in blocking pores to prevent substantial migration of moisture.

4.4.4 General Assessment

For Dravo process dewatering/forming, results indicate that the Laval and Albany oils are marginally satisfactory with respect to as-formed moisture content. Only the Albany oil provided adequate product strength in the dry condition.

On this basis, it is considered that only the Albany oil would provide acceptable product properties at dosages equivalent to petroleum oils. Since the Albany product is prepared by a pressure hydrogenation process, it is likely to have a significantly higher manufacturing cost than pyrolysis products and thus be less competitive with petroleum binders.

4.5 NRC OIL AGGLOMERATION

Results of oil agglomeration tests on biomass conversion products were uniformly negative. Agglomerates suitable for screen dewatering, using a 0.5mm screen, could not be obtained in useful yield at dosages up to 20% (vs dry coal) using either the Albany or Laval oils.

For the Laval oil at the highest (20%) level tested, a weak floc was recovered at 42% yield but microscopic examination revealed no evidence of the spherical micropellet structure which is readily attained with much lower doses of petroleum oil. Also, the moisture content of recovered material was formidably high (47%).

Results of tests on the Albany oil by NRC staff (Appendix 1) tend to confirm the poor results obtained in this work, in that - with Albany oil alone - ". . . The coal slurry flocculated . . . but would not form large, well defined agglomerates". In the NRC test dilution of the Albany oil with xylene to a 50/50 wt% solution allowed separation of flocs, but not formation of discrete agglomerates. In general, results of this work suggest that the biomass derived materials are unsuitable for oil agglomeration as normally practiced.

4.6 FROTH FLOTATION

Since the spherical agglomeration testwork produced no useful results, a limited number of froth flotation tests were done with the Albany and Laval oils.

Results are presented in Table 7.

At a 2% addition level, both biomass products acted as moderately strong but non-selective collectors. The Cold Lake pitch showed similar properties whereas No. 2 oil at 0.25% addition level allowed significant rejection of clay under the dilute (5%) pulp conditions used.

Moisture contents of filter cakes prepared from biomass oil froth concentrates were higher than that for the No. 2 oil, which is compatible with a floc structure rather than compact microagglomerates.

4.7 PIN AGGLOMERATION

Since testwork on the Dravo and particularly the oil agglomeration methods failed to reveal attractive applications for biomass derived oils, a limited program of tests on dry coal fines was undertaken. Results of a concurrent project indicated that the -150μ fraction of thermal dryer "dust" could be effectively eliminated by pin agglomeration ("turbulation") with ~6% No. 6 oil addition. This amount of oil is, however, prohibitively expensive and adds an unacceptable amount of volatile matter to the final (coking) coal product. Comparative tests on sulphite lignin indicated satisfactory agglomeration at a (potentially economic) 1% dosage level, but this resulted in unacceptable loss of coking properties.

The (non-selective) flocculating action of biomass oils at 2% addition level in flotation tests suggested the possibility of relatively strong binding action providing that excessive water is not present.

Results of pin agglomeration tests are presented in Table 8. For the Laval oil at 3%, (added as a 30% emulsion) results are comparable to those obtained with oil at 5-6% and lignin at 1%. The Albany oil did not perform as well, probably due to the fact that even at 100°C it was difficult to obtain a highly dispersed aqueous emulsion of this material. Thus the binding action was (presumably) limited by the surface area of semi-solid dispersed particles.

Preliminary assessment of coking properties of microagglomerates formed with Laval oil (Table 9) indicate that the material does not significantly affect the parameters tested. This is a potentially attractive result since it suggests that the biomass product is sufficiently modified to be acceptable as a binder for coking coals whereas lignin and cellulose fibre (raw biomass) both have significant deleterious effects on coke strength.

4.8 GENERAL ASSESSMENT OF TEST DATA

Results of this study suggest that the biomass oils tested are not suitable for oil agglomeration of fine coal and - in the best case - are only marginally competitive with petroleum oil for Dravo filtration/forming.

For microagglomeration of thermally dried coal, emulsified Laval oil appears to be a technically acceptable binder at a dosage level below that required for oil.

4.8.1 Preliminary Economic Analysis

Use of Laval oil (emulsified) as a binder for thermally dried coal is the only application identified in which a biomass derived product has equivalent or better properties than petroleum oil and could therefore be priced at oil equivalency or higher. In this application, sulphite lignin actually provides the price limitation (if its effect on coking can be discounted), since it can be delivered to southeast B.C. mines for ~$(Can) 0.50-0.60/Kg of contained dry solids. At a 1% addition level, this establishes an effective dose cost of $5-6/tonne which could allow pricing of Laval oil at $0.17-0.20/Kg. This is roughly eqivalent to current prices (weight basis) for No. 6 fuel oil.

If this application were developed, a market for roughly 9,000 tonnes/year of biomass oil would be available for each million tonnes of coal production. Current southeast B.C. annual coking coal production is in the order of 9 million tonnes/year. Thus, capture of any significant share of this potential market for biomass oil binder would provide an outlet for oil product from a reasonably sized (100-200 tpd wood waste feed) biomass conversion plant. It is also worthwhile to note that the price of lignin is strongly related to fossil fuel prices due to the energy costs of water evaporation for recovery from relatively dilute pulping solution. Thus any significant upward movement in oil costs may improve the competitive position of biomass derived oils.

4.8.2 Requirement for Further Testwork

There are some significant limitations to the general applicability of current results.

First, the Laval oil was described [9] as the heaviest fraction of product from a batch test. Other product fractions may, therefore, be less modified and less compatible with maintenance of coal coking properties.

The Laval oil was derived from poplar (hardwood), while wood waste in southeast B.C. is almost exclusively from softwood. Although it is likely that the pyrolysis products from softwood will have similar properties, this requires experimental confirmation.

Finally, it is not known at this time whether the Laval or other process(es) could generate product at $0.17-0.20/Kg. This is quite critical to consideration of an application to coal which is a low value commodity itself and cannot be priced to reflect the cost of expensive additives.

If it can be shown that Laval oil or - by inference - Waterloo oil which is also a pyrolysis product, could be produced economically for a (current dollar) price in the $0.20/Kg range at a scale of roughly 50-100 tonnes/day then further work on the identified application would be justified.

An appropriate program would include:

- testing the full range of product fractions to determine which have an acceptable combination of binding strength and compatibility with coking properties.

- testing (for promising fractions or blends) of the full range of coking properties and small scale oven tests to demonstrate acceptable product properties.

It should be noted that a test program of this type would require at least kilogram quantities of each product fraction. Judging by the difficulty in obtaining useful sized samples for the current program, a significant commitment of funds to produce the required materials (from softwood waste) would also be necessary.

5.0 CONCLUSIONS

Results of the study are considered to support the following conclusions:

- biomass derived "oils" are not suitable substitutes for petroleum oils in oil agglomeration of coal fines where selectivity and cohesion are required.

- biomass derived "oils" do not offer any technical advantages over petroleum oils in the Dravo filtration/forming process for coal fines.

- the Laval product tested is compatible with coking coal properties at up to 3 wt% addition level.

- emulsified Laval product is a technically acceptable binder for pin agglomeration of dry coal fines.

6.0 RECOMMENDATIONS

The identified potential market for biomass oil would require that the product be available at a price of about $0.20/Kg.

On this basis, it is recommended that staff of the Bioenergy Development Program review available data on the Laval and Waterloo pyrolysis processes to determine whether there is a realistic prospect of economic biomass oil production at this price, and a scale of 50-100 tonnes/day.

If such a production cost is feasible, consideration should be given to further technical studies on the specific application as outlined in Section 4.8.1.

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TABLE 1: PROXIMATE ANALYSES AND COKING DATA FOR TEST COALS

	BYRON CREEK	FORDING	VESTAR GREENHILLS*
Volatile Matter	23.1	24.2	26.1
Fixed Carbon	67.7	67.4	66.8
Ash	9.2	8.4	7.1
Free Swelling Index	1	6	8
Coke Stability	38-40	46-48	48-50

* Dryer Dust

TABLE 2: SIEVE SIZE ANALYSES OF TEST COALS

APERTURE (Micron)	(Mesh)	BYRON CREEK	FORDING	VESTAR GREENHILLS
		wt% Retained		
1410	12	0	0	0.5
500	32	0	0	18.1
250	60	0	0	22.5
150	100	<0.1	7.1	23.1
200	74	22.1	46.2	20.3
325	44	45.8	35.9	7.2
PAN		32.0	10.8	8.3

TABLE 3: PROPERTIES OF BIOMASS CONVERSION PRODUCTS

		ALBANY TR-12	LAVAL	B.C. RESEARCH Steam Pyrolysis (350°C)	B.C. RESEARCH Pressure Thermolysis (300°C)
Pour Point (°C)		60	30	>100	>100
Moisture (wt%)		3	4	6	<1
TGA Simulated Distillation, °C	25%	262	175	140	155
	50%	330	270	290	310
	75%	420	365	ND	ND
TGA Carbonization (%)					
Distillables		67.2	48.0	42.1	52.9
Volatiles		12.7	33.2	31.7	25.1
Char		17.8	17.9	25.1	27.3
Ash		2.3	0.9	1.1	0.5
Elemental Analysis (% Moisture Free)	C	73.1	66.4	57.1	64.1
	H	7.5	6.2	7.4	7.3
	N	0.1	0.2	0.1	<0.1
	O	14.7	25.9	35.9	32.6
THF Solubles (%)		95.4	97.2	56.2	45.1

TABLE 4: PROPERTIES OF PETROLEUM BASE BINDERS

		NO. 2 FUEL OIL	NO. 6 FUEL OIL	COLD LAKE PITCH
Pour Point (°C)		ND	5	45
TGA Simulated				
Distillation °C	25%	110	260	ND
	50%	140	310	ND
	75%	160	ND	ND
TGA Carbonization (%)				
Volatiles		>99	87	79
Fixed Carbon		<1	12	19
Ash		<1	1	2
Elemental Analysis (%)	C	85.5	86.0	83.2
	H	14.1	12.9	10.1
	N	<0.1	0.3	0.8
	O	0.3	<0.1	0.3
	S	0.2	0.6	5.5

TABLE 5: RESULTS OF DEWATERING TESTS WITH DRAVO PRESSURE FILTRATION/FORMING

BINDER	WT%*	FIBRE (wt%)*	AGGLOMERATE MOISTURE (% as Formed)
None	-	1	21
No. 6 Oil	-	0	19
	3	0	16.5
	5	0	16.5
	1	1	18
	3	1	17
	5	1	17
Pitch	1	0	20
	3	0	19
	5	0	19
	1	1	20.7
	3	1	20.3
	5	1	19.9
Laval Oil	1	0	No Agglomerate
	1	1	28.5
	3	1	22.1
	5	1	20.6
Albany TR-12	1	0	23
	3	0	23
	5	0	24
	1	1	22
	3	1	21
	5	1	20.5
B.C. Research	1	0	No Agglomerate
Steam Pyrolysis	3	0	32
	5	0	30
	1	1	28
	3	1	28
	5	1	28

* vs dry Byron Creek coal.

TABLE 6: RESULTS OF DROP-SHATTER PRODUCT STABILITY TESTS ON DRAVO DEWATERED PRODUCT

BINDER (3%)*	MOISTURE (%)	SIZE DISTRIBUTION, wt%				
		+6mm	6 x 2mm	2 x 0.5mm	0.5 x 0.15mm	-0.15mm
No. 6 Oil	17	45	20	13	15	7
Pitch	20.4	51	18	12	10	9
Albany TR-12	21	41	22	15	14	8
Laval Oil	22.1	39	23	16	12	10
B.C. Research Pyrolysate	28	35	18	23	10	12
No. 6 Oil	Oven Dry	39	28	11	12	10
Pitch	Oven Dry	36	33	12	10	9
Albany TR-12	Oven Dry	31	29	18	12	10
Laval Oil	Oven Dry	22	20	18	14	26
B.C. Research Steam Pyrolysate	Oven Dry	17	15	13	11	44

*Dry basis on Byron Creek coal, tests with 1% fibre additive except as noted.

TABLE 7: RESULTS OF FROTH FLOTATION TESTS*

COLLECTOR (Kg/T)	FROTHER (g/T)	WT% (DRY)		ASH % (DRY)		FROTH FILTER CAKE MOISTURE (%)
		Froth	Tailing	Froth	Tailing	
No. 2 Fuel Oil (2.5)	MIBC (20)	97	3	7.6	33	26
No. 6 Fuel Oil (20)	MIBC (20)	99	1	8.2	25	22
Pitch (20)	MIBC	>99	<1	8.3	ND	27
Laval Oil (20)	Nil	96	4	8.3	10.1	35
Albany TR-12 (20)	Nil	80	20	8.3	8.5	38
B.C. Research Steam Pyrolysate (20)	Nil	>99	<1	8.3	ND	32

*Denver D-2 machine, 1L cell, 900 rpm, 5% solids (Fording coal) in pulp; all pulps conditioned and floated at 80°C except with No. 2 oil (ambient temperature).

TABLE 8: RESULTS OF PIN AGGLOMERATION TESTS*

BINDER	WT%	DRY PRODUCT SIZE DISTRIBUTION			
		+0.5mm	0.5 x 0.25	0.25 x 0.15	-0.15
None	0	18.3	22.8	23.1	35.8
No. 6 Oil	3	22.3	24.6	25.3	27.8
	6	26.9	30.9	33.8	8.4
Laval Oil	3**	27.3	32.1	32.5	8.1
Albany TR-12	3**	18.9	27.2	27.6	26.3
Sulphite Lignin (Orzan A)	1***	27.2	29.3	34.9	8.6

* Westar Greenhills Dryer Dust; 2 passes through laboratory turbulator.
** Added as 30% emulsion.
*** As 10% solution.

TABLE 9: COKING PROPERTIES OF MICROAGGLOMERATED COAL*

BINDER, %		FREE SWELLING INDEX	COKE STABILITY
None	0	8	48-50
No. 6 Oil	3	9	50-52
Laval Oil	3	8	48-50
Sulphite Lignin	1	5	38-40
Groundwood Fibre	1	6	38-40

*Westar Greenhills

APPENDIX 1

NRC OIL AGGLOMERATION REPORT

NRCC/CHEM
CNRC/CHIMIE

FACSIMILE MESSAGE

374

DATE: June 25/87

TO/A: R O McElroy, Ind. Chem Div., BC Research
c/o of IRAP/NRC office

FROM/DE: C E Capes
NRC Ottawa

FAX: 1-604-666-7035

Please forward

SUBJECT/SUJET:

Tests with PERC and Cold Lake Oils

# of pages: 9 (including this cover page)
nombre de pages:

If there is any problem receiving this message, please call Darlene at (613) 993-2455 or (613) 993-0198. Our fax number is 613-993-6392.

MESSAGE:

Enc'osed is a report on work Dick Coleman did here on the PERC and Cold Lake Oils. I believe it is self-explanatory. We would like to hear your reaction. It seems to be in line with your finding of low strength with the PERC as a binder in the "pucks".

I will be in Vancouver next week on some private holiday. Will call you Friday June 26 to discuss these results and possibly meeting with you next week? I will be seeing Wes Johnston in Victoria in PM of July 2.

SIGNATURE: Ed Capes.

Conseil national de recherches Canada
National Research Council Canada

DOSSIER N°
FILE NO

DATE
9 March 1987

A File
TO

Re: Oil Agglomeration Tests Using Heavy Oils Being Employed by B.C. Research for Pelletizing or Briquetting Beneficiated Coal

Two oil samples, PERC TR-12 and Cold Lake Pitch, and a small amount of beneficiated Byron Creek coal were received from B.C. Research at the request of C.E. Capes, to allow us to evaluate these oils as possible bridging liquids for coal beneficiation. Our sample number for these materials is 87-02.

The two oils were very viscous and had to be heated at 110°C in order for them to become fluid. Even at this elevated temperature they could not be handled in a syringe. The oils were added glob wise to samples of Byron Creek coal suspended in hot water, about 85°C, and thoroughly mixed in a Waring Blender. The coal slurry flocculated with both oils but would not form large, well defined agglomerates. The addition of a small amount of xylene to the PERC TR-12/coal flocs produced small more compact aggregates, but nothing resembling discrete agglomerates which could be separated by screening, regardless of the amount of oil and xylene added. The addition of a small amount of No. 2 fuel oil to the Cold Lake Pitch/coal flocs produced discrete agglomerates which could be readily separated by screening.

The PERC TR-12 was diluted to a 50/50 wt.% mixture with xylene. The resulting mixture had a specific gravity of 0.970 and a weight loss at 110°C of 53.73 wt.%. The calculated specific gravity of the pure oil is 1.08. Since PERC TR-12 would only dissolve in an aromatic oil like xylene, we assume it is highly aromatic and will have agglomeration properties similar to aromatic oils such as the coal tars.

The Cold Lake Pitch was diluted to a 50/50 wt.% mixture with No. 2 fuel oil. The resulting mixture had a specific gravity of 0.914 and a weight loss at 110°C of 52.47 wt.%. The calculated specific gravity of the pure oil is 1.01. Since Cold Lake Pitch dissolved in an aliphatic oil like No. 2 fuel oil, we assume it is highly aliphatic and will have agglomeration properties similar to aliphatic oils such as the NRC No. 6 fuel oil.

Oil agglomeration tests using the Breckenridge waste coal fines, sample 85-10, and the diluted heavy oils, sample 87-02, were conducted. Test results are shown in Tables 1 to 4. Tables 5 and 6 are results obtained previously for Coaltek/TVA using the Breckenridge waste coal fines and No. 2 fuel oil.

The PERC TR-12/xylene mixture produced flocs which were readily separated using the floc flotation separation method, however, it was not possible to grow these flocs into discrete agglomerates.

...../2

Perhaps a higher temperature even with this dilute mixture would help, but I think not. The flocs did form rapidly - 30 seconds high shear mixing was sufficient - indicating good oil dispersion and at least sufficient coal surface wetting to form flocs. Product ash values of about 30% were obtained with combustible recovery & yield values of 96 and 68%, respectively. These ash values are much higher than those obtained from either the Cold Lake Pitch/No. 2 fuel oil mixture or the No. 2 fuel oil, which are both aliphatic oils. This is in agreement with NRC generally accepted agglomeration principles. Aromatic oils are not selective with respect to ash rejection (1).

The Cold Lake Pitch/No. 2 fuel oil mixture produced flocs which were readily separated using the floc flotation separation method. These flocs grew readily into discrete agglomerates which could be separated from the tailings by screening, upon the addition of a sufficient amount of the Pitch/No. 2 mixture. Product ash values of about 16% were obtained with combustible recovery and yield values of about 93 and 65%, respectively. These ash values are slightly higher than those obtained previously with No. 2 oil. Again, this is in agreement with NRC generally accepted agglomeration principles. Higher viscosity aliphatic oils produce higher product ashes than lower viscosity aliphatic oils.

In all cases, a product wash reduces the ash content and yield, however, the combustible recovery remains unchanged, indicating the rejection of more mineral matter. The smallest rejection occurred with the aromatic agglomerated product.

The oils in order of descending beneficiation potential are: No. 2 fuel oil, a 50/50 wt.% mixture of Cold Lake Pitch in No. 2 fuel oil and a 50/50 wt.% mixture of PERC TR-12 in xylene.

R.D. Coleman

/sl

(1) "Selective Oil Agglomeration in Fine Coal Beneficiation", C. E. Capes and R. J. Germain, "Physical Cleaning of Coal", Y. A Liu, Ed., Marcel Dekker, New York, 1982.

TABLE 1

OIL AGGLOMERATION OF BRECKENRIDGE WASTE COAL WITH B.C. RESEARCH HEAVY OILS

UNWASHED

%OIL (D.B., feed)	%OIL (D.B., prod)	PRODUCT QUALITIES % ASH (D.B., prod)	MASS YIELD (%)	COMBUSTIBLE RECOVERY (%)	TAILINGS QUALITIES %ASH	ASH REJ. (%)	FEED ASH (calculated) (%)
.94	1.46	30.83	64.38	83.23	74.82	57.32	46.50
2.77	3.49	31.19	79.50	96.49	90.30	42.74	43.30
4.76	6.38	30.17	74.63	95.68	90.72	50.55	45.54
6.20	7.89	31.20	79.49	96.27	90.27	44.22	43.90
7.71	10.33	29.90	74.97	95.68	90.53	50.28	45.08
14.27	20.22	24.39	70.58	94.27	88.97	60.32	43.39

50/50 mixture of Perc TR-12/Xylene

TABLE 2

OIL AGGLOMERATION OF BRECKENRIDGE WASTE COAL

WITH B.C. RESEARCH HEAVY OILS

WASHED

%OIL (D.B., feed)	%OIL (D.B., prod)	PRODUCT QUALITIES % ASH (D.B., prod)	MASS YIELD (%)	COMBUSTIBLE RECOVERY (%)	TAILINGS QUALITIES %ASH	ASH REJ. (%)	FEED ASH (calculated) (%)
.94	2.31	20.54	40.82	60.69	64.49	81.98	46.55
2.77	4.00	24.65	69.24	92.16	85.58	60.67	43.39
4.76	7.07	24.23	67.32	93.89	89.85	64.29	45.68
6.70	8.86	24.26	69.90	94.74	90.23	61.56	44.12
7.71	11.47	23.50	67.28	94.13	90.20	65.12	45.84
14.27	21.94	19.62	65.04	92.91	88.58	70.82	43.73

50/50 mixture of Perc TR-12/Xylene

TABLE 3

OIL AGGLOMERATION OF BRECKENRIDGE WASTE COAL

WITH B.C. RESEARCH HEAVY OILS

UNWASHED

		PRODUCT QUALITIES			TAILINGS QUALITIES		FEED ASH
%OIL (D.B., Feed)	%OIL (D.B., prod)	% ASH (D.B., prod)	MASS YIELD (%)	COMBUSTIBLE RECOVERY (%)	%ASH	ASH REJ. (%)	(calculated) (%)
.22	.54	21.42	41.46	56.76	57.60	79.15	42.60
.45	.71	14.25	63.36	93.62	89.89	78.49	41.97
.84	1.34	16.67	62.30	92.87	89.43	76.45	44.10
2.55	3.96	15.79	64.21	93.58	89.63	75.38	42.32
3.96	5.26	43.10	75.39	94.90	90.63	40.70	54.72
4.16	6.61	15.89	62.83	93.47	90.06	77.02	43.46

50/50 mixture of Cold Lake Pitch/No.2 fuel oil

TABLE 4

OIL AGGLOMERATION OF BRECKENRIDGE WASTE COAL

WITH B.C. RESEARCH HEAVY OILS

WASHED

%OIL (D.B., Feed)	%OIL (D.B., prod)	PRODUCT QUALITIES % ASH (D.B., prod)	MASS YIELD (%)	COMBUSTIBLE RECOVERY (%)	TAILINGS QUALITIES %ASH	ASH REJ. (%)	FEED ASH (calculated) (%)
.22	.71	8.80	31.43	43.95	58.11	93.51	42.61
.45	.79	6.92	57.04	91.52	88.54	90.60	41.90
.84	1.50	8.69	55.83	91.24	88.92	89.01	44.13
2.55	4.82	9.28	58.89	92.58	89.59	87.08	42.30
3.96	9.75	10.30	40.65	81.74	86.27	92.44	55.39
4.16	7.19	10.13	57.81	92.07	89.39	86.56	43.57

50/50 mixture of Cold Lake Pitch/No.2 fuel oil

TABLE 5

BRECKENRIDGE CAMP #11 WASTE FINES
LABORATORY AGGLOMERATION W/ No. 2 FUEL OIL/FLOC RECOVERY/No WASH

DATE d/m/yr	SAMPLE No.	BOOK REF.	WEIGHT % OIL (d.b. Feed)	WEIGHT % OIL (d.b. Prod.)	PRODUCT % ASH(d.b.)	PRODUCT MASS YIELD %	PRODUCT COMB. REC %	PRODUCT SCREEN (mesh)	TAILS % ASH(d.b.)	TAILS ASH REJ %	% ASH in Feed	PULP DENSITY %
6/8/85	85-10	XIV 114	0	0	—	0	0	FLOC RECOVERY				
		126	0.42	0.86	11.67	48.40	89.16	"	89.93	89.15	52.05	9.88
		115	0.85	1.52	11.50	55.90	88.73	"	85.76	85.47	44.25	9.68
		116	1.61	2.72	11.42	59.32	91.38	"	87.81	84.06	42.50	10.18
		117	2.31	3.83	12.55	60.30	92.17	"	88.72	82.32	42.79	10.67
		118	3.04	5.45	11.71	55.77	90.17	"	87.86	85.61	45.39	10.81
		119	3.60	6.12	13.36	58.76	91.69	"	88.81	82.35	44.47	11.41
		120	4.58	7.56	12.24	60.69	93.23	"	90.17	82.68	42.88	10.74
		121	5.63	9.42	13.24	59.71	92.85	"	90.10	82.12	44.21	10.22
		122	5.75	9.09	12.78	63.27	94.09	"	90.57	80.45	41.35	11.42
		123	6.60	10.61	12.79	62.17	93.67	"	90.31	81.12	42.12	11.20
		124	7.17	11.26	13.11	63.76	94.00	"	90.27	79.69	41.12	11.44

TABLE 6

BRECKENRIDGE CAMP #11 WASTE FINES
LABORATORY AGGLOMERATION TESTS W No. 2 FUEL OIL/FLOC RECOVERY/WASH

DATE d/m/yr	SAMPLE No.	BOOK REF.	WEIGHT % OIL (d.b. Feed)	(d.b. Prod.)	PRODUCT % ASH(d.b.)	MASS YIELD %	COMB REC %	SCREEN (mesh)	TAILS % ASH(d.b.)	ASH REJ %	% ASH in Feed	PULP DENSITY %
			0	0	—	0	0	FLOC RECOVERY				
9/8/85	85-10											
		XIV -158	0.75	1.37	5.66	54.77	89.50	"	86.59	92.66	42.26	10.96
		-150	1.46	2.59	6.24	56.58	92.41	"	89.97	91.71	42.59	11.21
		-157	2.16	3.78	6.26	57.16	92.15	"	89.35	91.45	41.86	11.40
		-151	2.83	4.92	7.11	57.44	92.80	"	90.28	90.39	42.51	11.62
		-154	3.31	5.69	7.71	58.18	92.67	"	89.84	89.33	42.05	12.40
		-152	4.25	7.34	6.89	57.95	92.67	"	89.85	[illegible]44	41.77	11.58
		-155	5.11	8.76	7.69	58.40	92.98	"	90.[illegible]	89.31	42.02	11.24
		-153	5.66	9.69	7.86	58.46	93.00	"	[illegible]4	89.08	42.00	11.60
		-154	6.77	11.46	8.16	59.07	92.94	"	89.94	88.42	41.64	12.13
		-156	7.01	11.96	7.54	58.59	92.80	"	89.85	89.39	41.63	10.55

** TOTAL PAGE.09 **